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PATENT SPECIFICATION

- *No.* 266121 -

CLASS 12 *o.* GROUP 17

DR. ARNOLD VOSWINKEL IN BERLIN.

Method for the manufacture of glycol urethane derivatives.

Amendment to Patent 247270.

Patented in the German Empire on 25 June 1912.

Longest Duration: 18 October 1925.

Further tests have shown that for the reaction of the main patent bromine and chloride acetyl urethane can be used with good results in place of bromo-acyetyl; in particular the reaction is faster and smoother than with the method described in the main patent. In addition to the chloride acetyl urethane and bromine acetyl urethane already described in the literature, other halogen urethanes are manufactured and described below.

The exposure of carbamine-acidified methyl to chloride acetyl chloride results in chloride acetyl carbamine acid methyl ester, which melts at a temperature of 145 °C.

When carbamine acidified methyl is exposed to bromo-acetyl chloride or bromo-acetyl bromide instead of chloroacetyl chloride, the result is bromoacetylcarbamine acid-methyl ester, whose properties are close to those of the corresponding chlorinated ester, and which melts at 148°C. The chloroacetyl-carbamine acid isobutylester resulting from the exposure of carbamine acid isobutylester to chloroacetylchloride, melts at a temperature of 78°C, the similar bromo-acetylcarbamine acid isobutylester melts at 87 °C.

These halogen acyl-urethanes can also be generated by exposing the Na-urethanes to fatty-acid esters of halogen.

Based on the embodiment of the main patent, the reaction can be described by the following equation:

$$C_2H_5O \cdot CO \cdot NH \cdot CO - CH_2Br + NaCOO\,CH_3 = C_2H_5O \cdot CO \cdot NH \cdot CO - CH_2 \cdot OOC \cdot CH_3 + Na\,Br.$$

Embodiment 1.

210 parts of bromoacetylurethane and 136 parts of acetatic sodium bicarbonate are boiled with 1500 parts of a high-proof alcohol for 4 to 6 hours at the reflux condenser. After filtering off the result from the precipitated bromosodium, the alcohol is concentrated to a small volume and the reaction product is crystallized. The acetyl glycol-urethane forms clear needles, which melt at about 98 °C. It is also easily soluble in water as well as in alcohol.

Embodiment 2.

473 parts of bromoisovalerian-acidic sodium and 525 parts bromoacetylurethane are boiled with 2500 parts of high-proof alcohol according to embodiment 1, and processed. The bromoisovalerianyl-glycolylurethane forms small, clear needles, which melt at 103 °C. It is not easily soluble in water but is easily accepted by spirit and ether.

Embodiment 3.

160 parts salicyl-acidic sodium and 210 parts bromacetylurethane are boiled and processed with 1500 parts alcohol as in Embodiment 1. The salicylic acid glycolylurethane crystallizes as it cools down. It is then siphoned off and washed with water to remove the bromosodium. The solid melts at 146 °C, is not soluble in water and ether, but easily soluble in warm alcohol.

Embodiment 4.

203 g bromoisovalerian-acidic sodium bicarbonate and 800 g absolute alcohol are boiled with 151 g chloro-acetylcarbamine acid methylester 3 to 4 hours at the reflux condenser, the precipitating common salt is filtered out, and the concentration of the spirit base is slightly increased. From the concentrated base crystallizes the bromisovalerylglycolylcarbamine acid methylester and melts at 90 °C, it is soluble in spirit and ether but not soluble in water.

Embodiment 5.

160 parts salicyl-acidic sodium bicarbonate are boiled with 151 parts of chloracetylcarbamine acid methylester in approximately 1000 parts spirit for several hours, the base is evaporated, the common salt is removed and set aside for crystallization. The salicyl-acidic glycolylcarbamineacid-methylester is fairly easily soluble in water and alcohol, and melts at 150°C.

For the reactions above, benzene or toluene may also be used instead of alcohol.

PATENT CLAIM:

Modification of the process protected by patent 247270 for the creation of glycolic acid ureide, **wherein** bromo- or chloroacetyl urethanes and the salt of an organic acid are exposed to each other in the presence of suitable solvents in order to produce the glycolylurethanes.

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CERTIFICATE OF TRANSLATION

December 5, 2006

I, Angela Christie, hereby certify that I am competent in both English and German languages.

I further certify that under penalty of perjury translation of the aforementioned document:

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from the German language into the English language is accurate and correct to the best of my knowledge and proficiency.

Angela C. Christie

Professional Translator

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